FINAL

CHARACTERISTIC EDS SPECTRA FOR LIBBY-TYPE AMPHIBOLES

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INTRODUCTION

Work performed by the United States Geological Survey (Meeker et al. 2003) has shown that asbestos material from the vermiculite mine near Libby, Montana, is characterized as a friable amphibole that spans a range of mineral classes including mainly richterite, winchite, tremolite, and actinolite. Magnesio-arfvedsonite and magnesio-riebeckite were also detected, but could not be confirmed as being in a fibrous form.

For the purposes of exposure and risk assessment at the Libby site, EPA groups this series of related amphiboles into the heading "Libby Amphibole Asbestos" (LA), and does not believe it is important to further differentiate the specific mineralogical classes.

During analysis of a sample by transmission electron microscopy (TEM), the identification of a particle as LA is based on three criteria: morphology, x-ray diffraction pattern, and chemical composition. Chemical composition is evaluated using x-ray energy dispersive spectroscopy (EDS). Use of EDS to classify an amphibole particle as LA is complicated by a number of factors, including a) inherent variability in chemical composition of different LA particles, b) dependence of the spectrum on random variables such as particle thickness, orientation, and proximity to other particles, and c) variation between instruments in sensitivity to different elemental constituents. For these reasons, it is not possible to define a unique EDS spectrum for LA particles. However, it is possible to consolidate the EDS spectra obtained from the analysis of many different asbestos particles that are known to be LA and to characterize the average spectrum and the range of variability in the spectrum of the primary elemental constituents. These consolidated spectra serve as a useful tool in judging when an EDS spectrum is sufficiently similar to the spectra from known LA particles to support the assignment of the LA designation.

METHODS

Samples used for this study included three samples of asbestos-rich material collected by the USGS from the Libby mine, along with a NIST asbestos standard identified as tremolite. The sample numbers assigned to these samples are listed below:

Sample Number	Source	Primary Amphibole Components
SW[x]1201	Libby mine (site 20)	Tremolite, Actinolite
SW[x]1281	Libby mine (site 28)	Richterite, Winchite
SW[x]1231	Libby mine (site 23)	Tremolite, Actinolite, Richterite, Winchite
SW[x]1TR1	NIST	Tremolite

Note: [x] = variable integer used to indicate different aliquots (bottles) of the sample

These four samples were provided to each of the six analytical laboratories that provide analytical support to EPA at the Libby site. These laboratories are:

- EMSL Analytical, Inc., Westmont, NJ (EMSL-Westmont)
- EMSL Mobile Asbestos Laboratory, Libby, MT (EMSL-Mobile)
- Reservoirs Environmental Inc., Denver, CO (RESI)

- Material Analytical Services, Inc., Suwanee, GA (MAS)
- Batta Environmental Associates, Inc., Newark, DE (Batta)
- Hygeia Laboratories Inc., Sierra Madre, CA (Hygeia)

Each laboratory obtained an EDS spectrum from at least 25 amphibole asbestos particles from each of the four materials. Each spectrum was recorded over an interval of about 5 minutes in order to ensure that sufficient counts were obtained that the spectrum was statistically stable. No correction for background was performed. The descriptions of the instruments used by each laboratory to measure the EDS spectra are found in Table 1.

RESULTS

Appendix A presents the raw data collected by each laboratory, and Appendix B presents summary statistics.

Figure 1 presents a graphical summary of the data. In order to consolidate spectra across different spectra, each spectrum was normalized to a silicone peak height of 10. The results for all LA structures (samples SW0121, SW01281, and SW01231) are presented in one graph, and the results for the NIST tremolite sample are presented separately. The bars on each peak represent the range between the 5^{th} and the 95^{th} percentile values for each element.

DISCUSSION

As shown in Figure 1, LA particles are characterized by the presence of sodium, magnesium, potassium, calcium and iron. Aluminum is usually absent, but may occur at low levels in some structures. As noted, there is substantial variability between particles and between instruments, but the following peak magnitudes (relative to silicone) are typical:

	Typical Peak Magnitude		
Element	Mean	5th	95th
Na	0.36	0.15	0.64
Mg	3.08	2.47	3.49
Al	0.08	0.00	0.27
Si	10.00	10.00	10.00
K	0.25	0.06	0.53
Ca	1.81	0.96	2.63
Fe	1.08	0.57	1.88

It is important to stress that these typical averages and ranges should NOT be interpreted as absolute rules for classifying a particle as LA. Rather, as noted above, it must be understood that there is substantial variability in EDS spectra from LA particles, both within and between laboratories, and that authentic LA particles may sometimes result in spectra that fall outside the bounds above. However, it is thought that the bounds above will include the majority of all LA structures.

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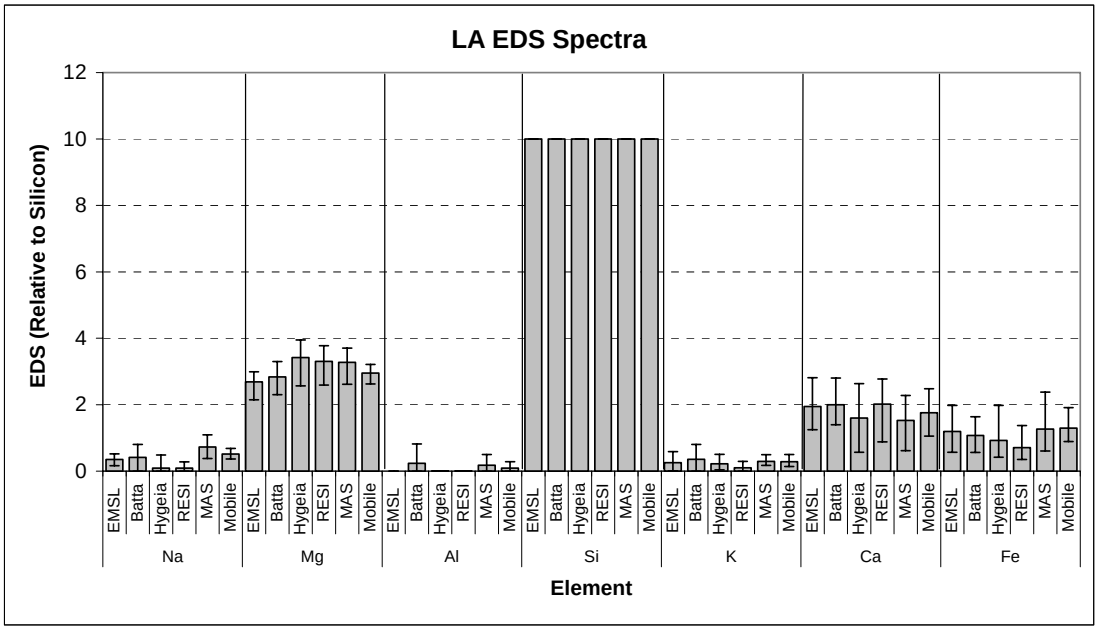
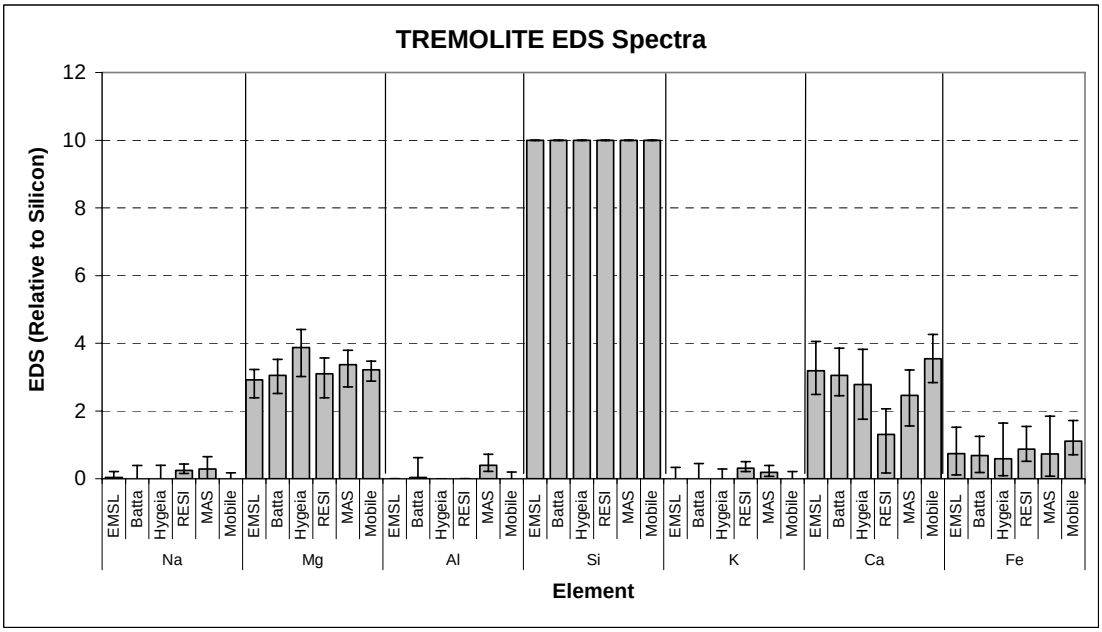
TABLE 1. TEM INSTRUMENT CHARACTERISTICS

Laboratory name	TEM Mfg.	TEM Model	Voltage (kV)	Window Type	Window Thickness	EDS Mfg.	EDS Model	eV per channel
Batta Laboratories, Inc.	JEOL	100CXII	100	Beryllium	8 um	Kevex	Delta 1	10
EMSL Analytical, Inc. (Westmont)	JEOL	100 CX II	100	Beryllium	8 um	PGT	XPJ-017-1038	10
Hygeia Laboratories Inc.	Hitachi	H-600	100	Beryllium	5.0 um	Kevex	Delta 1	10
Materials Analytical Services, Inc.	JEOL	1200 EX	100	Z-Max	Not specified	Noran	Z-Max 30	10
Materials Analytical Services, Inc.	JEOL	1200 EX	100	Beryllium	7.6 um	Noran	611D-3SST	10
EMSL Analytical Inc (Mobile Lab)	JEOL	100 CX II	100	Beryllium	8 um	PGT	XS16-J017	10
Reservoirs Environmental Inc.	JEOL	100 CX	100	Beryllium	8 um	Tracor	5502	10

FIGURE 1 EDS SPECTRA FOR LA AND TREMOLITE





Bar height indicates average and range (5^{th} to 95^{th} percentile) compared to silicon

APPENDIX A

Raw Data

APPENDIX B

Summary Statistics